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Experimental Plan to Investigate Critical R&D Issues of a Cryogenic Cold Trap

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Abstract

The first step in tritium extraction from the helium loops of a solid breeder blanket is the removal of HTO (and H_2O) in a cold trap operated with liquid nitrogen. For the operational conditions of the ITER blanket loops, several critical R&D issues have been identified. They will be investigated experimentally with a cold trap taken over from FZJ. The setup for these tests and the experimental plan are described. The results will be used for the construction of a new trap in which appropriate temperature profiles can be realized and which can be used also for tritiated gases.

Plan zur experimentellen Untersuchung von wichtigen Eigenschaften einer kryogenen Kaltfalle

Zusammenfassung

Der erste Prozeß-Schritt zur Tritium Extraktion aus den Helium-Kreisläufen eines Feststoff-Blankets besteht in der Abscheidung von H_2O/HTO in einer mit flüssigem Stickstoff betriebenen Kaltfalle. Bei den vorgesehenen Betriebsdaten der Blanketkreisläufe sind einige physikalische und technische Schwierigkeiten zu überwinden, die hier diskutiert und anhand von Experimenten mit einer vom FZJ übernommenen Kaltfalle untersucht werden sollen. Der Versuchsaufbau und die geplante Versuchsdurchführung werden beschrieben. Die Ergebnisse dienen der Konstruktion einer neuen Kaltfalle, in der sich geeignete Temperaturprofile einstellen lassen und die auch für den Betrieb mit tritierten Gasen benutzt werden kann.

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1. Introduction

During the past five years, several steps have been undertaken to develop a reliable method for tritium extraction from the purge gas of a ceramic breeder blanket:

- A literature study has been carried out to evaluate ten different process options published since 1988 /1/.
- On the basis of this evaluation, an additional procedure (a modification of an ANL concept /2/) has been proposed /3,4/ which is expected to have a good potential for reliable application to an ITER test blanket as well as to blankets of larger fusion machines, such as DEMO.
- This modified concept has been further developed and described in a contribution to the ITER Design Description Document prepared for the European Helium Cooled Pebble Bed (HCPB) test blanket /5/.
- Critical R&D issues of the concept have been discussed in a separate paper /6/. Together with the assessment of the process options, this paper was the starting point of the EU task TR6 entitled "Tritium Recovery from an ITER Test Blanket Module".

The objective of this task is to test and to optimize the process steps of a Tritium Extraction System (TES) with the help of the test facility PILATUS (*Pil*ot-*A*nlage zur *T*riti*u*m *S*eparation) in the Tritium Laboratory of Karlsruhe, TLK /7/. The max. gas flow rate in the facility will be about 6 times smaller than in the TES loop designed for the ITER Test Blanket Module (TBM).

As tritium will occur in the purge gas in the chemical forms of HT and HTO, two main process steps are foreseen:

- Removal of Q_2O (Q = H, D, T) with a cold trap at ≤ - 100°C,
- Removal of Q_2, N_2, and O_2 with a cryogenic molecular sieve bed at -195°C.

Several critical R&D issues of the concept are associated with the design of the cold trap and the measurement of very small humidity concentrations. This is evident from the operational conditions of the trap, as below:

Gas flow rates:	17	Nm³/h	for the ITER TBM
	1 - 3	Nm³/h	for the PILATUS tests
H_2O/HTO inlet concentrations:	10 - 50	ppmv	in both cases
H_2O/HTO outlet concentrations:	< 0.1	ppmv	in both cases

Cold trap experiments carried out with similar but less ambitious objectives in France at CEA /8/, in Italy at JRC /9/, and in Japan at JAERI /10/ have led to results which are important for the present investigations:

a) During the cool down of the gas in a cold trap operated with liquid nitrogen (LN_2) water vapour removal can be achieved by condensation on the inner walls of the trap as well as by trapping of ice aerosol particles on a filter.

b) Ice aerosol formation has two main disadvantages:
Very small particles (≤ 1 µm) can pass through the filter and leave the trap without being removed; larger particles are retained on the filter but cause clogging and an increasing drop of pressure.

c) To obtain a high removal efficiency, the formation of ice particles should be avoided; this can be done by cautious cool down of the gas, i.e. by avoiding fast under-cooling and critical supersaturation /11/ which give rise to spontaneous aerosol formation. The presence of particulate impurities in the gas should also be prevented because such impurities will act as condensation nuclei for aerosol formation. The most appropriate temperature gradient in the trap depends on the gas flow rate and on the humidity of the gas. It will be necessary to test each cold trap with different temperature gradients to optimize its behavior for a specific gas flow.

Instead of using these results for the construction of a new cold trap it was decided to carry out a series of pretests with an available cold trap. This trap was formerly designed for the removal of xenon from the dissolver off-gas of a reprocessing plant /12/. It was taken over from Forschungszentrum Juelich, FZJ.

A description of the trap is given in Chapter 2 of the present report. Following a specification of the pretest objectives (Chapter 3), the experimental setup is described in Chapter 4. The techniques to be applied to the quantitative assessment of the humidity are discussed in Chapter 5. Finally, the main steps of the experimental plan for the pretests are summarized in Chapter 6.

At present (November 1999), the experimental setup is being completed, and first functional tests have been started. The pretest results will be used for the construction of a new trap to be installed in the PILATUS facility.

2. Description of the FZJ Cold Trap

The FZJ cold trap consists of two precooling zones and a main freezing zone. Figure 1 shows the dismantled trap. The inner part (centre) is suspended from a flange where all gas inlets, outlets and electrical feed throughs pass through. Below the flange, there are the two pre-coolers and the main cooler which has 22 copper plates as precipitators for the condensed water vapour. The main cooler is enclosed in an additional container (right). Good temperature isolation is achieved using this second container and an isolating vacuum established between the inner and the outer container (left). Additional details of the trap are shown in Figs. 2 - 4.

Fig. 1: Dismantled FZJ Cold Trap

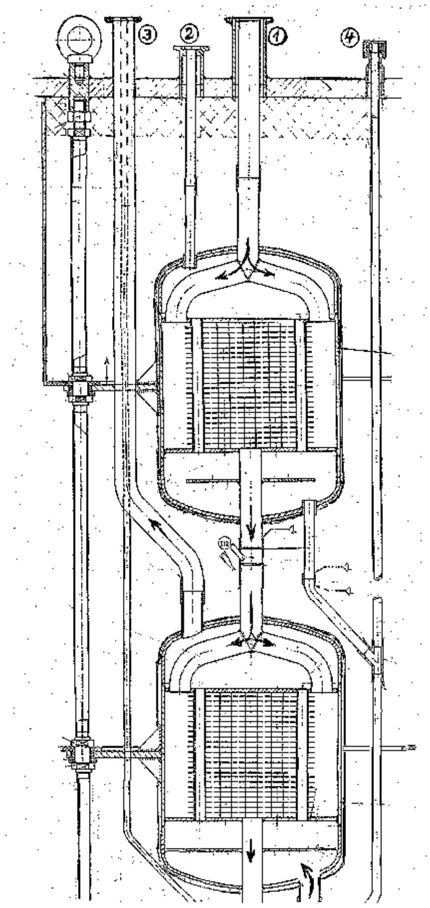

Fig. 2: Upper Part of the Trap

The test gas is introduced in the central flange No.1. It is precooled in the first zone by evaporated N_2 gas which leaves the trap through the flange No. 2. The test gas temperature is then further reduced in the second precooling zone by the cold test gas coming from the main cooling zone (not shown in this figure).
Label No. 3 represents two flanges placed at the same distance from the central flange: the main test gas outlet and the LN_2 inlet; the latter has a smaller diameter than the outlet tube of the test gas.
At flange No. 4, there is an outlet for test gas samples taken from single cooling plates of the main cooling zone (see Figs. 3 and 4).
The test gas temperature can be measured with a PT-100 element after each precooling zone.

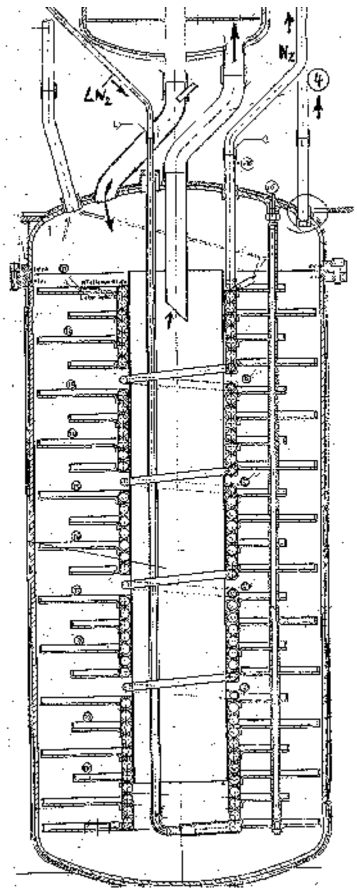

Fig. 3: Lower Part of the Trap

The main cooling zone is shown in Fig. 3. It contains the copper plates to precipitate water vapour, and a spiral tube through which the cooling medium is circulated. The 40 windings of the spiral tube and additional windings of 5 separate electrical heaters are mounted around a central cylinder.
LN_2 enters the spiral at the bottom of the trap, then it moves upwards until it is evaporated and further warmed up by the countercurrent of the test gas and by the electrical heaters. Thus, different temperature gradients can be established within the main cooling zone. The temperature profile is measured with 13 PT-100 elements attached to the copper plates at different levels.
The test gas entering the space between the inner and the outer cylinder from the top is carried along the copper plates. After being cooled down to approx. 80 K, the test gas is carried back through the inner cylinder to the second precooling zone.

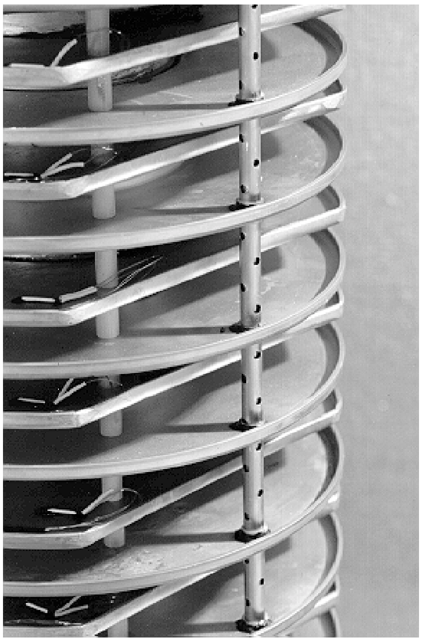

Fig. 4: Arrangement for Test Gas Sampling

As indicated above, it is possible to take gas samples from different horizontal levels within the main cooling zone. This is achieved using a perforated vertical tube in which a sampling tube is moved through flange No. 4 to the region of interest. The sampling tube is closed at its lower end; a hole serving as inlet for the gas sample is placed at a distance of 20 mm from the end.

The main geometrical and operational parameters of the trap are given in the following table:

Diameter:	350	mm
Total height:	1433	mm
Volume of the main freezing zone:	26	ltr
Surface area in the main freezing zone:	1.3	m^2
Heater power:	300	W
Throughput:	$\leq$ 10	Nm^3/h
Gas pressure:	0 - 8	bar abs.

3. Objectives of the Pretests

It is the primary objective of the pretests to study the removal efficiency of H_2O as a function of following parameters

- temperature profile in the cold trap,
- initial humidity of the test gas,
- gas flow rate.

Additional objectives are:

- to test several analytical methods to determine humidity concentrations in the range < 5 ppmv,
- to study the process of ice aerosol formation and the transport / deposition of these aerosols,
- to use the experimental results for the construction of an improved and – if possible – more compact cold trap to be utilized with tritiated gases in the PILATUS facility.

4. Experimental Setup

A flowsheet of the experimental setup is shown in Fig. 5.

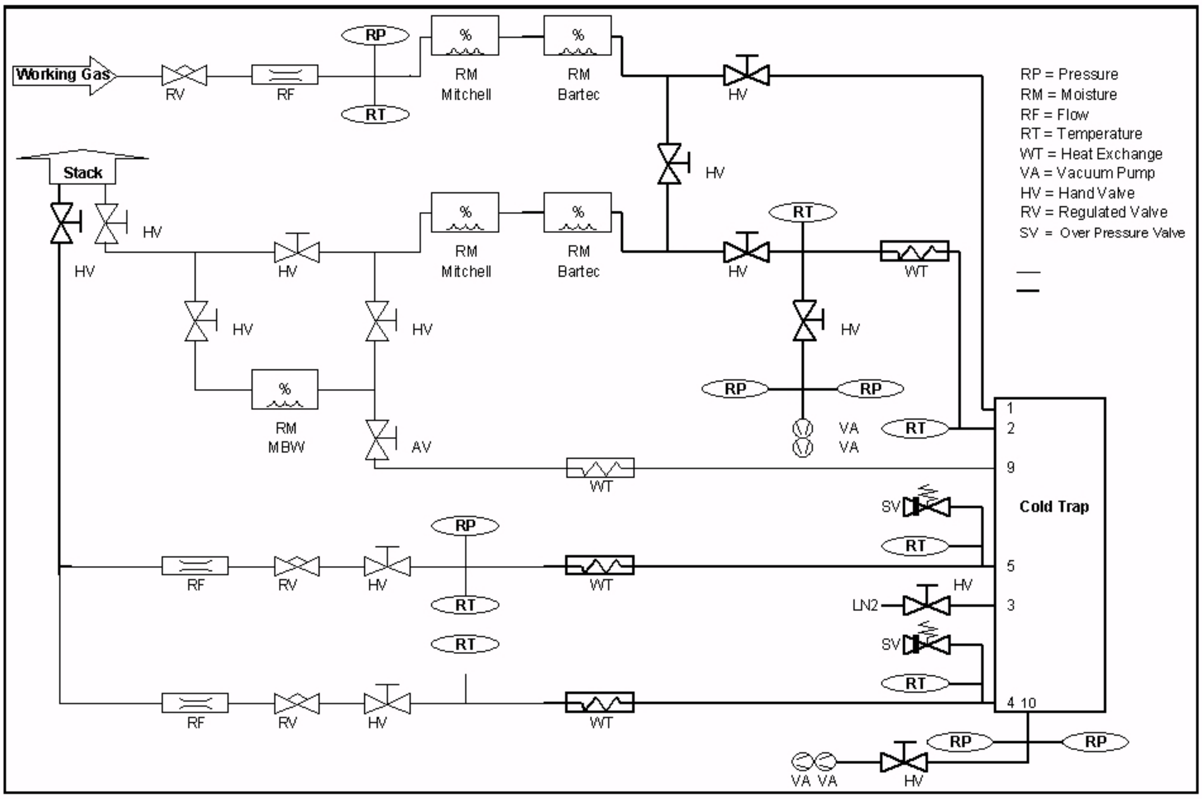

Fig. 5: Flowsheet of the Pretest Facility

The test gas (working gas) is taken from a steel cylinder containing helium with a premixed addition of 10 (20/50) ppmv of water vapour. The nominal gas composition can be determined with two different moisture analyzers supplied by Mitchell and by Bartec. Normally the gas is then delivered to the inlet of the cold trap (Port 1).
In special cases, e.g. to compare the response of different sensors, the cold trap can be bypassed; the gas is then delivered into the main exit line of the trap starting at Port 2. This line contains a second set of analyzers as described above. It also contains a heat exchanger (WT) to warm up the test gas to ambient temperature, plus a very sensitive optical dew point hygrometer made by MBW (Switzerland). This instrument is placed in a bypass to the main gas line because only small gas samples are needed for analysis. It is also available to measure the small gas samples taken from single areas of the main cooling zone leaving the trap at Port 9.
Port 3 is the inlet for LN_2, Port 5 is the main outlet for the evaporated nitrogen; it corresponds to flange No. 2 in Fig. 2. Port 4 is a second nitrogen exit not explained in the preceeding figures. It can be used to reduce the precooling intensity in the first precooling zone. In this case most of the evaporated N_2 is directly released after leaving the main cooling zone. Both N_2 exit ports are connected with over pressure valves set at 2 bar abs. and with heat exchangers to raise the gas temperature. Temperature control is performed with thermocouples up- and downstream of the heat exchangers. In addition, two pumping systems are available, one for the evacuation of the test gas loop, and one for an isolating vacuum which is controlled at Port 10. Electropolished tubes are used for all the test gas lines.

Instrumentation for Process Control (see Fig. 5):

- 2 Vacuum pumping systems (VA) with appropriate control instruments, one system to evacuate and to remove residual moisture from the test gas loop and one system for the isolation vacuum inside the outer shell of the cold trap
- 3 Flowmeters (RF), one for the test gas and two for the N_2 off-gas lines
- 3 Regulation valves (RV), one for the test gas and two for N_2 off-gas lines; these valves can be used to vary the temperature inside the cold trap
- 2 Pressure indicators (RP), one for the test gas and one for the N_2 off-gas (the pressure in the two N_2 lines is identical)
- 7 Temperature indicators (RT), one for the test gas at the inlet side of the cold trap (needed for the humidity indication by the Michell hygrometer), and 2 x 3 to indicate the temperature up- and downstream of the heat exchangers (WT) at the cold trap outlet Ports 2, 4 and 5
- 2 + 13 PT-100 elements in the interior of the cold trap (see Figs. 2/3).

All electrical signals of the corresponding sensors / indicators are sent to a data processing system (µ-MUSYCS-T, manufactured by Additive Soft- and Hardware GmbH, D) for further evaluation and storage.

5. Humidity Measurement

The main analytical issue is the quantitative measurement of humidity concentrations in the ppm and sub-ppm range. As current experience in the TLK is not very advanced in this field, it was decided to apply three different physical methods and to extend the scope of the analytical techniques later if necessary.
Two detection systems have been selected for the measurement of the <u>inlet</u> concentration:

- Hygrophil F (Manufacturer: Bartec, D)
- Transmet Dew Point Transmitter (Manufacturer: Michell, UK)

The humidity concentration at the <u>outlet</u> of the trap is determined by using a second Michell system, a second Hygrophil sensor (if needed), and a

- Cooled Mirror Dewpointmeter K-1806/DP3-D (Manufacturer: MBW Electronic, CH)

<u>Hygrophil F</u> is a fiber optical hygrometer consisting of a separate sensor and an electronic unit. Both are connected by a two-lead fiber optical light guide.

Principle of operation:	Infrared light is sent from an optoelectronic emitter accommodated in the electronic unit via the light guide to the sensor. The spectral intensity distribution of the reflected light varies according to the amount of water molecules adsorbed on the micro-porous surface of the sensor. The reflected light is conducted through the second lead of the light guide and analyzed in a polychromator. The humidity concentration is then determined using the calibration data stored in the electronic unit.
Sensitivity range:	Dew point - 70°C .... + 10°C or 2.5 ppmv .... 12 000 ppmv

The Michell Transmet Transmitter system consists also of a separate sensor and an electronic unit.

Principle of operation: The sensor is a capacitance device. It is built up of conductive layers on top of a base ceramic tile. The capacitance changes when water vapour molecules penetrate into the ceramic. The humidity in the gas is derived from the measurement of this change.

Sensitivity range: Dew point -100°C ... +20°C or 0.014 ppmv ... 23 000 ppmv

According to the nominal sensitivity range, this sensor should be able to determine the humidity of the test gas at the inlet of the cold trap as well as at the outlet. It is suspected, however, that the response time of the sensor will be too long when decreasing humidities are to be measured in the sub-ppm range.

The Cooled Mirror Dewpointmeter is a hygrometer system where the sensor is contained in a relatively large cabinet (W x H x D = 548 x 473 x 435 mm) because the operation of the sensor requires several additional installations such as a cooling circuit, a 3-stage Peltier element, a lamp with stabilized power supply and a photo-electric cell.

Principle of operation: A mirror is cooled by a compressor cooling system and three additional Peltier elements until a dew film of a defined thickness has been formed. The corresponding temperature of the mirror is measured and transformed into a signal indicating the dew point.

Sensitivity range: Dew point – 90°C .... +20°C or 0.1 ppmv .... 23 000 ppmv

6. Experimental Plan

6.1 Functional Tests

After the PILATUS pretest facility is assemled functional tests will be carried out to examine the properties of the loop and of all process components. The tests will include:

- Leak tests of the piping system and the cold trap;
- Confirmatory tests of the flowmeters, pressure and temperature indicators, and the humidity sensors. These tests will mainly examine the mechanical properties and the electronical connections without giving results on the validity of the calibrations.
- Cool down tests with LN_2 including tests of the PT-100 elements in the trap. The tests will give first results on the speed of cool down, the temperature profile and the time required to reach temperature equilibrium. Parameters: LN_2 inlet pressure, flow rate of the test gas (pure helium).
- Tests of the thermal isolation and the propagation of cold regions outside of the trap. It is especially important to control the temperatures in the cold exit lines of the trap to avoid condensed water on the piping system and to keep the operating temperature of the electronic devices (e.g. flowmeters) close to room temperature.

6.2 Test Program

The test program consists of two main parts:

- Investigation of all relevant properties of the cold trap which must be known to carry out successful humidity removal tests;
- Measurement of the humidity removal efficiency for various operational conditions.

Investigation of the Relevant Cold Trap Properties

It is known that water condensation will occur on the internal surfaces of the trap as well as by spontaneous formation of water droplets in the atmosphere which are later transformed into ice aerosol particles. A certain fraction of these aerosol particles will be transported through the trap without being deposited. As a consequence, they will lead to an undesirable residual humidity in the gas leaving the trap.

It is the aim of the investigations to determine the <u>optimum temperature profiles</u> which lead to a predominantly surface condensation. These profiles will depend on the nature of the gas (heat capacity, flow rate) and on the operational conditions of the cold trap. They are characterized by the temperature measurments of the 15 PT-100 elements (see Chapter 4) and recorded as a function of time.

The profiles will be investigated with pure helium as test gas and as a function of the following parameters:

- LN_2 supply at gas flow rates of e.g. 0, 0.5, and 2.0 Nm³/h;
- Supplementary heating by using 1,2 ... or 5 electrical heaters (rf. text to Fig.3), gas flow rates in similar steps as above;
- Reduction of the test gas precooling by the evaporated N_2 in the first precooling zone (achieved by releasing a fraction or all evaporated N_2 through Port 4 as described in Chapter 4).

Measurement of the Humidity Removal Efficiency

Humidity removal tests will start with temperature profiles which allow a slow cool down of the test gas, especially in the temperature range near and below the dew point where saturation and supersaturation effects may lead to enhanced aerosol formation. Additional profiles causing a greater cool down velocity will then be investigated.

The residual humidity will be measured as a function of the following parameters:

- Gas flow rate (0.3 – 3 Nm³/h, initial humidity 10 vpm, various temperature profiles)
- Initial H_2O concentration (10 – 50 ppmv, 1 Nm³/h, various temperature profiles)

In each case, the removal efficiency will be determined from the ratio of the humidity concentrations at the outlet and the inlet of the cold trap.

Acknowledgement

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